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## **ORIGINAL SPECIFICATION**

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Method and system for decontaminating a clean-room

The present invention relates to a method for decontaminating

5 a clean-room, in which the clean-room is supplied with gaseous H<sub>2</sub>O<sub>2</sub>, and also relates to a system for decontaminating a clean-room, which system comprises an H<sub>2</sub>O<sub>2</sub> supply device for supplying the clean-room with H<sub>2</sub>O<sub>2</sub>.

10 In the context of this description and the patent claims, decontamination is also taken to mean sterilization and disinfection. Clean-room means all rooms which can be tightly sealed, for example isolators, locks, microbiological safety workbenches, sterilizers and transfer containers for the 15 pharmaceutical industry, cosmetics, chemistry, food technology, electronics, nuclear industry, experimental animal husbandry, medicine, etc.

In food technology, hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) has already been 20 used for many years in liquid form as a decontamination agent. Since, in high concentrations (> 3%) it can act corrosively on various materials, it has not immediately found an opening in clean-room technology. Since the beginning of the 1980s, the microbiocidal properties of H<sub>2</sub>O<sub>2</sub> 25 at small concentrations have been intensively studied. It was found that H<sub>2</sub>O<sub>2</sub> in vapor form, even at low concentration (100-5000 ppm), can destroy not only bacteria and spores thereof, but also fungi, yeasts and viruses. Since H<sub>2</sub>O<sub>2</sub> does not act selectively, it is widely usable. In addition to 30 formalin and peracetic acid, H<sub>2</sub>O<sub>2</sub> has therefore been used in the past for rapid and safe decontamination of clean-rooms.

A system for decontaminating a clean-room which comprises an H<sub>2</sub>O<sub>2</sub> supply device for supplying the clean-room with H<sub>2</sub>O<sub>2</sub> is disclosed, for example, in CH-A-689 178. This system, in one 5 variant, has an evaporator unit, an H<sub>2</sub>O<sub>2</sub> supply vessel and a conveying device for conveying liquid H<sub>2</sub>O<sub>2</sub> from the H<sub>2</sub>O<sub>2</sub> supply vessel to the evaporator unit. The H<sub>2</sub>O<sub>2</sub> supply vessel is disposed outside the clean-room and is connected via flexible tubing to the evaporator unit which is disposed 10 within the clean-room. To supply the clean-room with H<sub>2</sub>O<sub>2</sub>, liquid H<sub>2</sub>O<sub>2</sub> is conveyed from the H<sub>2</sub>O<sub>2</sub> supply vessel to the evaporator unit and there evaporated, after which it is distributed in the clean-room. This is performed until the decontamination concentration is achieved. This is, in the 15 case of H<sub>2</sub>O<sub>2</sub>, approximately 100-5000 ppm and is usually maintained for approximately from 10 to 120 minutes. After the decontamination, an exhaust air flap valve is opened and the H<sub>2</sub>O<sub>2</sub>-containing exhaust air is flushed out of the clean-room and passed via an exhaust air channel, in which case, to 20 reduce the emissions, a catalyst can be present in the exhaust air channel, which catalyst decomposes the H<sub>2</sub>O<sub>2</sub>, for example into H<sub>2</sub>O and O<sub>2</sub>. Recirculation of the H<sub>2</sub>O<sub>2</sub>-supplied air over a catalyst is also known.

25 It is disadvantageous in this decontamination method that the excess H<sub>2</sub>O<sub>2</sub>, if it is broken down, is broken down using a catalyst. In order to achieve sufficiently rapid breakdown times, relatively large amounts of catalyst are required, which is very expensive. A further disadvantage is that the 30 catalysts used need to be regenerated. Furthermore, any H<sub>2</sub>O<sub>2</sub> breakdown does not proceed until outside the clean-room, that is to say the H<sub>2</sub>O<sub>2</sub> must first be flushed out of the clean-

room. Complete flushing of H<sub>2</sub>O<sub>2</sub> out of the clean-room is relatively difficult, since it partially condenses in the clean-room and adheres to surfaces. In order that a desired residual concentration of usually from 5 to 0.05 ppm can be 5 achieved, generally a flushing time of at least one hour is required, even if the clean-room is heated to evaporate the condensed H<sub>2</sub>O<sub>2</sub>.

In view of the disadvantages of the previously known above 10 described methods and systems for decontaminating a clean-room, the following object underlies the invention. A method and a system for decontaminating a clean-room are to be provided, which are to make possible, in the least costly manner possible, a decontamination using H<sub>2</sub>O<sub>2</sub> and then to 15 achieve as rapidly as possible the desired residual H<sub>2</sub>O<sub>2</sub> concentration.

This object is achieved by the inventive method and the inventive system, as are defined in the independent patent 20 claims 1 and 8. Patent claim 15 relates to an inventive H<sub>2</sub>O<sub>2</sub> breakdown device for such a system. Preferred variants result from the dependent patent claims.

The essence of the invention is that in a method for 25 decontaminating a clean-room, the clean-room is supplied with gaseous H<sub>2</sub>O<sub>2</sub> and, at a later timepoint, H<sub>2</sub>O<sub>2</sub> still present in the clean-room is chemically broken down without catalyst by supplying at least one gaseous agent which reacts with the H<sub>2</sub>O<sub>2</sub>.

30

Owing to the fact that the excess H<sub>2</sub>O<sub>2</sub>, that is to say the H<sub>2</sub>O<sub>2</sub> which has not reacted with other materials in the clean-

room during the decontamination is broken down in the clean-room itself, it need not be flushed out completely from the clean-room first. Furthermore, H<sub>2</sub>O<sub>2</sub> which is condensed in the clean-room need not be evaporated first, as a result of which  
5 a heating of the clean-room can be dispensed with. The time for flushing out the exhaust air can thus be reduced to a few minutes. Therefore, cycle times of less than 60 minutes for decontaminating and flushing out are achievable, which corresponds to a considerable reduction compared with the  
10 prior art.

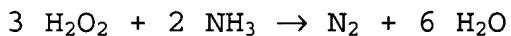
Owing to the gas form of the agent, this is distributed well in the clean-room and also comes into contact with the H<sub>2</sub>O<sub>2</sub> which is condensed on surfaces, so that it rapidly reacts  
15 with the H<sub>2</sub>O<sub>2</sub> and breaks this down.

Finally, owing to the fact that the H<sub>2</sub>O<sub>2</sub> is chemically broken down without catalyst, no expensive catalysts are required for the breakdown of H<sub>2</sub>O<sub>2</sub> in the clean-room or in the exhaust  
20 air.

Advantageously, H<sub>2</sub>O<sub>2</sub> residues in a product situated in the clean-room are subsequently broken down on the product in a targeted manner. This is, for example, of importance when a  
25 lower H<sub>2</sub>O<sub>2</sub> concentration is wanted for the product than is present in the clean-room after the H<sub>2</sub>O<sub>2</sub> breakdown, and can be performed with the same agents.

Preferably, the at least one gaseous agent is metered in such  
30 a manner that after the chemical breakdown of the H<sub>2</sub>O<sub>2</sub> at most 1 ppm of H<sub>2</sub>O<sub>2</sub> still remains in the clean-room. Such a residual concentration is not a problem.

Preferably, the at least one gaseous agent comprises ammonia ( $\text{NH}_3$ ). This reacts with the  $\text{H}_2\text{O}_2$  as follows:



5 The ammonia reduces the  $\text{H}_2\text{O}_2$ , with  $\text{N}_2$  and water which is primarily in gaseous form being exclusively formed, that is to say harmless environmentally compatible reaction products. Since no precipitate is formed, these breakdown products can be flushed out of the clean-room without a problem into the  
10 exhaust air channel which need not conform to any special requirements with regard to chemical resistance. Furthermore, the exhaust air which can also comprise ammonia residues, can be discharged into the open without further treatment, since in addition to the breakdown products, the ammonia itself is  
15 also environmentally compatible.

Ammonia under usual ambient conditions, is a gas, it is easy to meter and is freely available on the market. The conventional quality ( $> 99.7\%$ ) is sufficient for the  
20 inventive application. Furthermore, only small amounts of ammonia are required, that is to say about 0.5 l of  $\text{NH}_3$  gas per g of pure  $\text{H}_2\text{O}_2$ . The amount of  $\text{H}_2\text{O}_2$  and ammonia used obviously depends on the volume of the clean-room, and can therefore differ widely. The space and power requirements for  
25 storing and introducing the ammonia into the clean-room are small. Overall, therefore the use of ammonia is significantly cheaper than the use of catalysts, in particular in procurement, but also in use.

30 Furthermore, ammonia has the advantage that, like  $\text{H}_2\text{O}_2$ , it has a high affinity to water, and is readily soluble therein.

Condensed H<sub>2</sub>O<sub>2</sub> takes up NH<sub>3</sub> gas very well and is rapidly broken down.

5 A further advantage of ammonia is that it can be used very well even in large clean-rooms.

Furthermore, in the case of optimum use of ammonia, it is not necessary to flush out the clean-room, since the resultant atmosphere in the clean-room corresponds to the desired 10 conditions.

15 Ammonia generally reacts with H<sub>2</sub>O<sub>2</sub> very rapidly. Practical experiments have found that, at 25-35°C, the reaction time is about 1-2 minutes. Since any interfering residual products will be present in gas form, they can also be rapidly flushed out from the clean-room. The cycle time for decontamination of the clean-room, the breakdown of the H<sub>2</sub>O<sub>2</sub>, and a flushing of the clean-room can be reduced to less than 60 minutes.

20 An advantage of ammonia is that it is environmentally compatible and the MAC value (maximum workplace concentration) is 50 ppm, which is significantly higher in comparison with H<sub>2</sub>O<sub>2</sub>. Ammonia residues are therefore less of a problem than H<sub>2</sub>O<sub>2</sub> residues. Furthermore, the odor of 25 ammonia is characteristic and gives a warning. Ammonia gas is therefore also used, for example, to test for leaks of the isolator comprising the clean-room, and any gloves which are present. These tests can be carried out in the inventive method at the end of the cycle directly before flushing out 30 the clean-room.

Control of the ammonia introduction is simple. It can be

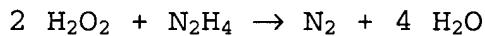
based on detecting an excess of ammonia or of H<sub>2</sub>O<sub>2</sub> in the clean-room using chemical indicators or sensors.

Preferably, ammonia is introduced into the clean-room in 5 excess, so that the breakdown reaction takes place rapidly and as completely as possible.

A disadvantage of ammonia is that it is flammable. However, the concentration which is required in the inventive method 10 is low and the ammonia is largely immediately broken down by the H<sub>2</sub>O<sub>2</sub>. Only a possible ammonia excess is critical. This is therefore advantageously kept so low that the ignition limit of 15% is not reached. The metering is such that the ammonia excess is at most 500 ppm.

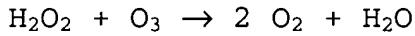
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As an alternative to, or in combination with, ammonia, hydrazine (N<sub>2</sub>H<sub>4</sub>) can be used as gaseous agent. This reacts with the H<sub>2</sub>O<sub>2</sub> as follows:



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The at least one gaseous agent can also comprise ozone (O<sub>3</sub>). This reacts with the H<sub>2</sub>O<sub>2</sub> as follows:



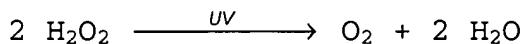
25 Ozone in the inventive method is not used to accelerate the sterilization, but to break down the H<sub>2</sub>O<sub>2</sub>.

The use of gaseous hydrazine or ozone to break down the H<sub>2</sub>O<sub>2</sub> is associated with similar advantages as the use of ammonia.

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In addition, the H<sub>2</sub>O<sub>2</sub> still present can be photochemically broken down by UV radiation. This usually takes place as

follows:



5 The UV light is preferably generated in the clean-room by a UV lamp disposed in the clean-room. It preferably has a wavelength of 254 nm.

The inventive system for decontaminating a clean-room  
10 comprises an  $\text{H}_2\text{O}_2$  supply device for supplying the clean-room with  $\text{H}_2\text{O}_2$  and an  $\text{H}_2\text{O}_2$  breakdown device for effecting chemical breakdown of  $\text{H}_2\text{O}_2$  without catalyst in the clean-room, which has means for introducing into the clean-room at least one gaseous agent, in particular ammonia, hydrazine or ozone.  
15 This system makes it possible to carry out the above mentioned inventive method which is associated with the advantages described.

In a preferred variant, the means for introducing at least one gaseous agent comprise a supply vessel charged with gaseous agent, for example a gas bottle, or a generator for producing gaseous agent, a gas line from the supply vessel or the generator to the clean-room, and a valve for regulating the amount of gaseous agent flowing through the gas line. The  
25 amount of the gaseous agent introduced into the clean-room can thus be regulated by means of the valve. In addition, gas cartridges can also be used which comprise the required amount of gaseous agent. A valve and a control device can then be omitted.

30

In an advantageous variant, the  $\text{H}_2\text{O}_2$  breakdown device additionally has means for generating UV light in the clean-

room. These means comprise, for example, a UV lamp which generates UV light within the clean-room. Such UV lamps are part of the prior art.

5 Advantageously, the inventive system has a sensor for measuring the concentration of the gaseous agent in the clean-room, the measured values of which serve to control the H<sub>2</sub>O<sub>2</sub> breakdown device. If an excess of gaseous agent is measured which is not broken down by reaction with H<sub>2</sub>O<sub>2</sub>, the  
10 introduction of gaseous agent into the clean-room is usually stopped.

Instead of the quantitative sensor mentioned, a qualitative indicator, for example color indicator, is also conceivable.  
15 The breakdown process can also be controlled manually in this manner.

Alternatively or in addition, the inventive system has a sensor for measuring the H<sub>2</sub>O<sub>2</sub> concentration in the clean-  
20 room, the measured values of which serve to control the H<sub>2</sub>O<sub>2</sub> breakdown device. If the sensor measures an H<sub>2</sub>O<sub>2</sub> concentration in the clean-room which is less than the sought-after residual concentration, for example 1 ppm, the H<sub>2</sub>O<sub>2</sub> breakdown no longer needs to be advanced. This means  
25 that no additional gaseous agent needs to be introduced into the clean-room, or no additional UV light needs to be generated in the clean-room.

For open-loop control or closed-loop control of the H<sub>2</sub>O<sub>2</sub> supply device, and of the H<sub>2</sub>O<sub>2</sub> breakdown device, preferably separate open-loop control and closed-loop control devices are provided which makes possible subsequent installation of

the H<sub>2</sub>O<sub>2</sub> breakdown device into an existing system having H<sub>2</sub>O<sub>2</sub> supply device.

5 The H<sub>2</sub>O<sub>2</sub> breakdown device can either be constructed as a separate device which, independently of the H<sub>2</sub>O<sub>2</sub> supply device, introduces gaseous agent into the clean-room, or generates it in this, or it and the H<sub>2</sub>O<sub>2</sub> supply device can be integrated into a periphery of the clean-room. In new decontamination apparatuses, generally, the integration of 10 the H<sub>2</sub>O<sub>2</sub> breakdown device and the H<sub>2</sub>O<sub>2</sub> supply device into the periphery of the clean-room is preferred, while it is simpler to retrofit existing decontamination apparatuses with a separate H<sub>2</sub>O<sub>2</sub> breakdown device.

15 The inventive system for decontaminating a clean-room is described in more detail hereinafter with reference to the accompanying drawings on the basis of two example embodiments. In the drawings:

20 Fig. 1 diagrammatically shows a first example embodiment of the inventive system having a separate H<sub>2</sub>O<sub>2</sub> breakdown device; and

25 Fig. 2 diagrammatically shows a second example embodiment of the inventive system having an H<sub>2</sub>O<sub>2</sub> supply device and H<sub>2</sub>O<sub>2</sub> breakdown device integrated into a periphery of the clean-room.

30 In the first example embodiment shown in Fig. 1 of an inventive system for decontaminating a clean-room 1, an H<sub>2</sub>O<sub>2</sub> supply device 2 is disposed outside a periphery 3 of the clean-room 1. An open-loop control and close-loop control

device 31 controls the conditions in clean-room 1, in particular the pressure relationships and the air conditions. The H<sub>2</sub>O<sub>2</sub> supply device 2 comprises, for example, as described in CH-A-689 178 at least one liquid-H<sub>2</sub>O<sub>2</sub>-filled H<sub>2</sub>O<sub>2</sub> supply vessel, at least one evaporator unit in the form of a heating plate for vaporizing the H<sub>2</sub>O<sub>2</sub> and at least one H<sub>2</sub>O<sub>2</sub> line between the at least one H<sub>2</sub>O<sub>2</sub> supply vessel and the at least one heating plate. The at least one heating plate is disposed in the clean-room 1, so that the H<sub>2</sub>O<sub>2</sub> which is fed from the 10 at least one H<sub>2</sub>O<sub>2</sub> supply vessel via the at least one H<sub>2</sub>O<sub>2</sub> line is vaporized directly in the clean-room 1 on the at least one heating plate. The supply of H<sub>2</sub>O<sub>2</sub> to the clean-room 1 is controlled by an open-loop control and closed-loop control device 21 which preferably comprises a stored-programmable 15 control. Usually, sufficient H<sub>2</sub>O<sub>2</sub> is vaporized in the clean-room 1 so that in clean-room 1 an H<sub>2</sub>O<sub>2</sub> concentration of approximately 100-5000 ppm is present for approximately from 10 to 120 minutes.

20 After the decontamination with H<sub>2</sub>O<sub>2</sub>, the H<sub>2</sub>O<sub>2</sub> still present in the clean-room 1, that is to say the H<sub>2</sub>O<sub>2</sub> which has not reacted and has not been consumed, is broken down using a gaseous agent which is introduced into the clean-room 1 via a gas line 13. As gaseous agent, use is preferably made of 25 either ammonia, hydrazine or ozone.

For this purpose, the system has a separately constructed H<sub>2</sub>O<sub>2</sub> breakdown device 10 which comprises a supply vessel 11 in which the gaseous agent is stored. The stock of gaseous 30 agent in the supply vessel 11 is monitored by a control unit 14. The gaseous agent stored in the supply vessel 11 passes into the clean-room 1 via the gas line 13, in which case one

or more nozzles can be provided at the clean-room-side end of the gas line 13, which nozzles distribute the gaseous agent in the clean-room 1. In the gas line 13, there is disposed a valve 12 with which the amount of the gaseous agent 5 introduced into the clean-room 1 can be introduced under open-loop or closed-loop control. The valve 12 is controlled via an open-loop control and closed-loop control device 15 which is connected to a sensor 4 for measuring the concentration of the gaseous agent and to a sensor 5 for 10 measuring the  $H_2O_2$  concentration. The sensors 4 and 5 are disposed in the clean-room 1 and measure the concentration of the gaseous agent and the  $H_2O_2$  concentration in the clean-room 1.

15 Depending on the values measured by the sensors 4 and 5, more or less gaseous agent is fed to the clean-room 1. Generally, a small excess of gaseous agent is introduced into the clean-room 1, so that the  $H_2O_2$  is broken down rapidly and as completely as possible.

20 After the breakdown of the  $H_2O_2$ , in the clean-room 1 the air exchange is ensured again, in which case for this purpose in a known manner a feed air channel, a feed air flap valve, an exhaust air flap valve and an exhaust air channel can be 25 provided. The system can in addition have further elements which are known from systems for decontaminating a clean-room of the prior art.

30 In the second embodiment example shown in Fig. 2 of an inventive system for decontaminating a clean-room 101, the  $H_2O_2$  breakdown device and the  $H_2O_2$  supply device 102 are integrated into the periphery 103 of the clean-room 101. The

H<sub>2</sub>O<sub>2</sub> breakdown device comprises, instead of a supply vessel for gaseous agent, a gas generator 111 which generates the gaseous agent directly. The generator 111 is controlled by an open-loop control unit 114. The gaseous agent generated is  
5 fed via a gas line 113 to the clean-room 101, the amount of agent fed being introduced under open-loop or closed-loop control via a valve 112 disposed in the gas line 113. The valve 112 is controlled by an open-loop and closed-loop control device 115 which is connected to a sensor 104 for  
10 measuring the concentration of the gaseous agent and to a sensor 105 for measuring the H<sub>2</sub>O<sub>2</sub> concentration. The sensors 104 and 105 are disposed in the clean-room 101 and measure the concentration of the gaseous agent and the H<sub>2</sub>O<sub>2</sub> concentration in the clean-room 101.

15 The open-loop control and closed-loop control device 115 is also connected to the control unit 114 and via this ensures that gaseous agent is generated or not in correspondence with the measured values of the sensors 104 and 105.

20 As in the first example embodiment, the clean-room 101 is supplied with H<sub>2</sub>O<sub>2</sub> under open-loop control and closed-loop control by an open-loop control and closed-loop control device 121 which preferably comprises a stored-programmable  
25 control. By means of an open-loop control and closed-loop control device 131, the conditions in the clean-room 101 are subjected to open-loop control and closed-loop control, in particular the pressure relationships and the air conditions. The open-loop control and closed-loop control device 121 is  
30 here connected via the open-loop control and closed-loop control device 131 to the open-loop control and closed-loop control device 115, so that the measured values of the

sensors 104 and 105 can also be used for the open-loop control of the H<sub>2</sub>O<sub>2</sub> supply.

Further, that said with reference to the first example  
5 embodiment applies.

Further structural variations can be implemented to the above described systems for decontaminating a clean-room. It may be further explicitly stated here that the H<sub>2</sub>O<sub>2</sub> supply device can also be constructed differently than described. For  
10 example, H<sub>2</sub>O<sub>2</sub> which is already gaseous could be introduced from outside into the clean-room 1 or 101. In principle, all H<sub>2</sub>O<sub>2</sub> supply devices of the prior art are conceivable.